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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/DK91/00030 (22) International Filing Date: 1 February 1991 (01.02.91) (30) Priority data: 269/90 1 February 1990 (01.02.90) DK (71) Applicant (for all designated States except US): ROCK- WOOL INTERNATIONAL A/S (DK/DK); Hovedgad- en 501, DK-2640 Hedehusene (DK). (72) Inventor; and (75) Inventor/Applicant (for US only): MOGENSEN, Gurli (DK/DK); Egevej 78, Vemmedrup, DK-4632 Bjæverskov (DK). (74) Agent: LEHMANN & REE; Frederiksberg Alle 26, DK- 1820 Frederiksberg C (DK).		(81) Designated States: AT, AT (European patent), AU, BB, BE (European patent), BF (OAPI patent), BG, BJ (OAPI patent), BR, CA, CF (OAPI patent), CG (OAPI patent), CH, CH (European patent), CM (OAPI patent), DE, DE (European patent), DK, DK (European patent), ES, ES (European patent), FI, FR (European patent), GA (OAPI patent), GB, GB (European patent), GR (Euro- pean patent), HU, IT (European patent), JP, KP, KR, LK, LU, LU (European patent), MC, MG, ML (OAPI patent), MR (OAPI patent), MW, NL, NL (European patent), NO, RO, SD, SE, SE (European patent), SN (OAPI patent), SU, TD (OAPI patent), TG (OAPI pa- tent), US. Published <i>With international search report.</i>
(54) Title: MINERAL FIBRES (57) Abstract The invention relates to high-temperature resistant mineral fibres having the following composition: SiO ₂ : 34-42 % by weight; Al ₂ O ₃ : 31-38 by weight; TiO ₂ : 0-1 by weight; FeO: 2.5-7.5 by weight; CaO: 17-21 by weight; MgO: 0.5-4.3 by weight; Na ₂ O: 0.3-1.5 by weight; K ₂ O: 0.1-2.3 by weight.		

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Mineral fibres

The present invention relates to mineral fibres which are resistant at high temperatures, and in particular high-temperature resistant mineral fibres for use in thermal insulation, e.g. in energy intensive furnaces.

Fireproof ceramic fibres are known which consist substantially of a major amount of SiO_2 and Al_2O_3 and optionally a minor amount of a further component and which has a maximum temperature of use (MTU) of about 1500°C or more, cf. US patent specifications Nos. 4,240,833, 2,674,539 and 2,710,261, DE laid open patent applications Nos. 1,075,295 and 1,596,842 and NO laid open patent application No. 145,467. Such fibres are manufactured from a melt composed of a mixture of SiO_2 and Al_2O_3 , and since this mixture does not melt until at a temperature of $1600-1900^\circ\text{C}$, the formation of the melt requires an electric furnace whereby the costs associated with the manufacture of said fibres become relatively high.

Mineral fibres is another kind of fibres and they consist of a mixture of the following oxides: SiO_2 , Al_2O_3 , FeO or Fe_2O_3 , CaO , MgO and R_2O wherein R is Na and K. Examples of such mineral fibres are rock wool fibres which typically have an MTU of up to 750°C and which may be manufactured at relatively low costs in coke heated shaft furnaces.

There is a need to provide low-cost mineral fibres having a somewhat higher MTU than ordinary rock wool fibres, viz. having an MTU of up to 850°C .

DK patent application No. 5453/87 discloses inorganic fibres having an MTU of up to 815°C and having the following composition:

SiO_2 :	55-64 % by weight
Al_2O_3 :	0-10 -
CaO :	29-44 -
MgO :	0.1-30 -

Also SE patent application No. 8406571-3 describes inorganic fibres

having a higher MTU than that of ordinary rock wool fibres, viz. an MTU of up to 800°C. These known fibres are manufactured from a chlorite-containing raw material which contains chlorite in an amount of more than 35%, preferably 40-45%, and has an iron oxide content of between 5 and 20%, preferably between 10 and 15%. The known fibres which may for instance have the following composition:

	SiO ₂ :	40.9%
	Al ₂ O ₃ :	14.7%
10	FeO:	5.3%
	Fe ₂ O ₃ :	8.3%
	CaO:	10.2%
	MgO:	6.8%
	K ₂ O+Na ₂ O:	3.3%
15	MnO:	0.2%
	TiO ₂ :	3.0%

suffer from the drawback that the colour of the fibres is very dark due to the relatively high iron oxide content.

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It is the object of the present invention to provide mineral fibres having an MTU which is substantially higher than the MTU of conventional rock wool fibres and which can be manufactured in conventional plants for the manufacture of rock wool fibres and which are not associated with the above mentioned drawback.

25

This object is obtained with the mineral fibres of the invention, said fibres being characterized in having the following composition:

30	SiO ₂ :	34-42 % by weight
	Al ₂ O ₃ :	31-38 -
	TiO ₂ :	0-1 -
	FeO:	2.5-7.5 -
	CaO:	17-21 -
35	MgO:	0.5-4.3 -
	Na ₂ O:	0.3-1.5 -
	K ₂ O:	0.1-2.3 -

This composition is characterized in having a high content of SiO₂

and Al_2O_3 which provides an increased temperature resistance. The FeO content ensures that a sufficient amount of seed crystal forming agents is present, which is essential to the behaviour of the fibres at temperatures above the crystallization temperature. FeO and CaO reduces the viscosity to a suitable level. The MgO content is kept at a low level as the CaO content is high; thereby a melting temperature minimum which occurs when the MgO and CaO contents are within the same range is avoided.

10 The improved high-temperature properties obtained with the mineral fibres according to the invention will appear from the drawing which shows the setting of mats made from mineral fibres of the invention (curve A), of conventional rock wool fibres (curve B) and of glass fibres (curve C) as a function of the temperature.

15 As will appear from the drawing the fibre product according to the invention is not only proof against a temperature which is about $100^\circ C$ higher than that of a corresponding product of rock wool fibres, but the setting is also substantially less (about 30% relative to about 45%) at temperatures above the maximum temperature of use. It is noted that the relevant fibre product according to the invention has the following composition:

	SiO_2 :	36.8 % by weight	
25	Al_2O_3 :	33.9	-
	TiO_2 :	0.88	-
	FeO:	4.4	-
	CaO:	19.5	-
	MgO:	2.6	-
30	Na_2O :	0.5	
	K_2O :	1.7	

The following is a particularly preferred composition for the mineral fibres according to the invention:

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SiO_2 :	36-40 % by weight	
Al_2O_3 :	33-35	-
TiO_2 :	0.6-0.9	-
FeO:	3.5-6.5	-

CaO: 18-20 -
MgO: 1.8-2.7 -
Na₂O: 0.4-0.6 -
K₂O: 1.5-2.1 -

5

The mineral fibres of the invention can be manufactured in a coke heated cupola furnace using a charge of e.g. 25 % by weight of lime and 75 % by weight of briquettes. The briquettes are preferably manufactured of kaoline as matrix (60 % by weight) added with
10 bauxite (20 % by weight) to raise the aluminium content, and iron silicate (20 % by weight) to raise the iron content.

The kaoline briquettes melt at relatively low temperatures. First the iron silicate melts, then the kaoline, and finally this melt
15 dissolves the bauxite. The final melt is formed by further dissolution of the lime in the charge whereby a suitable viscosity for the removal from the cupola furnace and the subsequent spinning process is obtained.

20 When the briquettes are melted in a coke heated cupola furnace, 13.7 % by weight of coke is preferably used (relative to the weight of briquettes and lime) and air is supplied through the tuyeres of the furnace at a rate of 80-96 m³/min. to obtain a melting temperature of 1565-1605°C. The melt which is removed through the melt outlet of
25 the furnace may be supplied to a conventional rock wool fibre spinning apparatus and converted to a fibre web therein.

The invention will now be described in further detail with reference to the following example.

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Example

A series of fibre samples were manufactured in the process described above. Thereby fibre samples having the following composition were
35 obtained.

Sample No.		1	2	3	4
5	SiO ₂ , % by weight	39.8	38.8	36.8	36.8
	Al ₂ O ₃ , "	34.4	33.9	33.9	34.9
	TiO ₂ , "	0.65	0.67	0.88	0.85
	FeO, "	2.5	3.3	4.4	4.2
	CaO, "	18.7	19.5	19.5	19.3
10	MgO, "	2.05	1.9	2.6	2.0
	Na ₂ O, "	0.48	0.45	0.50	0.48
	K ₂ O, "	2.0	1.9	1.7	1.7
	MnO, "	0.05	0.05	0.07	0.06

The fibre samples manufactured were subjected to a heat stability test (AGI Q132, cf. DIN 52271 A81) at a heating rate of 5°K/min and at a load of 1.0 kN/m².

Hereby the following maximum temperatures of use (MTU) were determined:

Sample No.	MTU
1	>850°C (at a specific weight of 206 kg/m ³)
2	818°C (at a specific weight of 91 kg/m ³)
3	815°C (at a specific weight of 120 kg/m ³)
4	821°C (at a specific weight of 111 kg/m ³)

C l a i m s

1. High-temperature resistant mineral fibres, characterized in having the following composition:

5

SiO₂: 34-42 % by weight

Al₂O₃: 31-38 -

TiO₂: 0-1 -

FeO: 2.5-7.5 -

10

CaO: 17-21 -

MgO: 0.5-4.3 -

Na₂O: 0.3-1.5 -

K₂O: 0.1-2.3 -

- 15 2. High-temperature resistant mineral fibres according to claim 1, characterized in having the following composition:

SiO₂: 36-40 % by weight

Al₂O₃: 33-35 -

20

TiO₂: 0.6-0.9 -

FeO: 3.5-6.5 -

CaO: 18-20 -

MgO: 1.8-2.7 -

Na₂O: 0.4-0.6 -

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K₂O: 1.5-2.1 -

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INTERNATIONAL SEARCH REPORT

International Application No. PCT/DK 91/00030

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶ According to International Patent Classification (IPC) or to both National Classification and IPC IPC5: C 03 C 18/06		
II. FIELDS SEARCHED Minimum Documentation Searched ⁷		
Classification System ⁸	Classification Symbols	
IPC5	C 03 G; C 04 B	
Documentation Searched other than Minimum Documentation to the extent that such Documents are included in Fields Searched ⁸		
SE, DK, FI, NO classes: as above		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹	Citation of Document ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
A	B; 145467 (JOHNS-MANVILLE CORPORATION) 21 December 1981, (cited in the description) --	1-2
A	A; 181624 (GRÜNZWEIG & HARTMANN AG) 13 August 1970, see the whole document -- -----	1-2
* Special categories of cited documents:¹⁰ "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step "Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "A" document member of the same patent family		
IV. CERTIFICATION		
Date of the International Completion of the International Search	Date of Mailing of this International Search Report	
26th April 1991	1991 -04- 30	
International Searching Authority	Signature of Authorized Officer	
SWEDISH PATENT OFFICE	May Hallne	

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO. PCT/DK 91/00030**

This annex lists the patent family members relating to the patent documents cited in the above-mentioned International search report. The members are as contained in the Swedish Patent Office EDP file on **91-02-28**.
The Swedish Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
NO-B- 145467	81-12-21	NONE	
DE-A- 1815244	70-08-13	FR-A-B- 2030094	70-10-30
		GB-A- 1282945	72-07-25
		US-A- 3679443	72-07-25